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Immediate and Delayed Repair Bond Strength of a New Ormocer Resin Restorative Material as a Function of Mechanical and Chemical Surface Conditioning Methods

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Short Title: *Repair Bond Strength of Ormocer Material*

Abstract

This study evaluated the μ-shear repair bond strength (μSBS) of a new ormocer restorative material as a function of repair time and repair protocol. Ormocer discs (N=140) (Admira Fusion, Voco) were prepared and divided into 14 groups: Factor 1: Bonding protocol (No Conditioning, Admira Bond, Futurabond M+, Silane/Admira bond, Silane/Futurabond M+, Ceramic repair system, Silane/Cimara bond) and Factor 2: Repair procedure time (immediate versus delayed). Each disc received two ormocer micro-cylinders. Half of the disks were tested immediately (24 h) and the other half after 6 months water storage. Shear test was run at crosshead speed of 0.5mm/min. Debonded specimens were evaluated for failure mode and SEM analysis was performed. Data were analyzed using two-Way ANOVA and Tukey`s tests (alpha=0.05). Both the repair time and the surface conditioning methods showed a significant effect on the repair μSBS (MPa) of the ormocer material (p=0.000). When immediate repair strengths were considered, all repair protocols tested (10.5±2.1±16.3-2.9) reached the mean bond achieved based on oxygen-inhibited layer only (no conditioning) (10.8±2.4 MPa). Futurabond M+ (13.9±3.4) and Silane/Cimara adhesives (16.3±2.9) showed significantly higher μSBS compared to that of the control group (p=0.001 and p=0.000, respectively). For the delayed repair, all bonding protocols showed significant decrease, where non-conditioned (5±1.7) followed by Admira Bond (7.1±1.7) groups, showed significantly lower values compared to those of the other protocols (9.2±2.2-10.4±2.9) (p=0.000 and p=0.000, respectively). Failure modes were predominantly adhesive type (immediate:95% and delayed:90%). No cohesive failures were observed either in the substrate or in the repair material.

Keywords: Adhesion; Bonding; Microshear; Ormocer restorative material; Repair bond strength.

1. Introduction

Resin composite restorations represent a unique class of dental biomaterials, as they restore dental tissues esthetically and functionally in a minimal invasive fashion. [1,2] For this reason, at the present time, almost more than half of the posterior cavities are restored with direct resin composite materials. [1]

Resin based materials are in continuous development regarding the chemical composition and polymerization methods in an attempt to overcome the inherent polymerization shrinkage of the material, low wear resistance and fracture toughness to improve their performance and longevity. [1-3] In this regard, Ormocer resin composite, which is an acronym for organically modified ceramics, is a class of direct bonded resin composites that was lunched in the market to decrease the polymerization shrinkage, increase wear resistance and enhance biocompatibility of resin based materials. [3-6] Despite these improvements, composite restorations may fail due to secondary caries, ditching at the margins, discoloration, delamination or fracture [4,5,7] For such reasons, decision making between total replacements of the defective restorations versus repair is controversial. Replacement of defective restoration could lead to increase in tooth tissue loss due to the difficulty in removing the tooth colored adhesive restoration without harming the sound dental tissue, increase chair time and cost. [7,8] On the other hand, in minimal invasive dentistry, repair was considered a more conservative approach that protects sound tooth structure, decreases pulpal injury, decreases the chair time and cost [4,5,7,8]. Accordingly, many dental schools today teach the concept of repairing defective resin restorations based on successful clinical and laboratory outcomes. [7]

Many factors affect the bond strength between the prepolymerized resin composite and the newly added composite layer. Among these factors, material compositions, surface conditioning methods in the form of either chemical, mechanical or combination of both, the use of silane coupling agents and repair time, either immediate or delayed, could all affect the adhesion in repair attempts. [4,5,7,8] To the best of our knowledge, there is limited data in the literature regarding the repair potential of ormocer material. [9,10]

The objective of this study therefore was to evaluate the repair bond strength of newly introduced ormocer material as a function of different bonding protocols and time. The null hypothesis tested was that neither repair protocols nor repair time would influence the repair bond strength of the ormocer material.

2. Materials and methods

An ormocer material (Admira Fusion, VOCO, Cuxhaven, Germany) was used as both substrate (shade A3.5) and repair material (shade A2). For the repair protocols, an etch-and-rinse adhesive, a self-etching adhesive, a silane primer and ceramic repair system were used. Materials, types, chemical compositions, batch numbers and manufacturer details are summarized in Table 1.

2.1. Specimen preparations

Ormocer discs (N=140) (thickness: 2 mm; diameter: 7mm) were used as substrates and pre-polymerized. The discs were divided into 14 experimental groups (n=10 discs) according to the two experimental factors evaluated in this study: Factor 1: Repair protocol (7 groups) and Factor 2: Repair time (2 groups). The allocation of experimental groups is illustrated in Fig 1.

Additional (n=10) discs were prepared (shade A3.5) and photo-polymerized to evaluate the repair strength on oxygen-inhibited layer without surface conditioning. Ten discs were prepared using split Teflon mold with central hole of 7 mm in diameter and 2 mm in height. The mold was rest on a glass slide covered with polyester strip (Stripmat, Polydentia, Mezzovico, Switzerland). The ormocer material (shade A3.5), were inserted inside the central hole of the mold and covered with a piece of Teflon tape. Another glass slide was used to gently press the material to extrude excess material and create a smooth flat surface. Both the glass slide and the Teflon tape were removed and each disc was photo-polymerized using a LED polymerization unit (Elipar, 3M ESPE, St. Paul, USA; light output: 1200 mW/cm^2) for 20 s. After polymerization, two polyethylene tubes (internal diameter: 1 mm; height: 0.7 mm) were filled with ormocer material (shade A2) and were adjusted over the surface. The material was packed inside the tubes, covered with polyester strip and gently pressed to extrude excess material. The material was then photo-polymerized for 20 s. The discs with their attached ormocer micro-cylinders were stored in distilled water for 24 h before testing.

2.2. Immediate repair procedure (Groups 1-7)

Discs (n=70, shade A3.5) were used as prepolymerized substrate ormocer (thickness: 2 mm; diameter; 7 mm). The discs were prepared in the same split teflon mold according to the manufacturer`s instructions. The mold was rest on a glass slide covered with polyester strip (Stripmat, Polydentia). The ormocer materials (shade A3.5), were inserted inside the central hole of the mold, covered with another polyester strip and gently pressed to extrude excess material using another glass slide. Each disc was photo-polymerized using a LED polymerization unit (Elipar, 3M ESPE; light output: 1200 mW/cm^2) for 20 s directly over the strip after removal of the glass slide. After polymerization, all prepared discs were immediately repaired with the repair ormocer material (shade A2) as follows:

Group 1: In this group, 10 discs were left intact and received no surface conditioning. Two polyethylene tubes (internal diameter: 1 mm; height: 0.7 mm) were filled with the ormocer material and the repair Ormocer (shade A2), and were adjusted over the intact unground surface. The material was packed inside the tubes, covered with polyester strip and gently pressed to extrude excess material and photo-polymerized for 20 s.

The remaining discs (n=60) were immediately wet ground with #600 grit silicon carbide paper for 10 s and were then repaired according to the following repair protocols:

Group 2: In this group, each disc was etched using 34.5% phosphoric acid for 15 s, rinsed for 20 s and air dried for 10 s. Adhesive resin (Admira bond, VOCO) was applied and left undisturbed for 30 s using micro-brush (Single Tim, VOCO). The adhesive resin was gently air dried for 5 s and photo-polymerized for 10 s. Two polyethylene tubes were filled with the ormocer material (shade A2), and were adjusted over the polymerized adhesive resin. The material was packed inside the tubes, excess material was extruded and photo-polymerized for 20 s as described in Group 1.

Group 3: Adhesive resin (Futurabond M+, VOCO) was applied and rubbed on each ground disc surface for 20 s according to the manufacturer`s instructions using the micro-brush. The adhesive was gently air-dried with compressed air for 5 s until there was no further movement of the adhesive over the surface and photo-polymerized for 10 s. Two polyethylene tubes were filled with the ormocer material (shade A2), and were adjusted on the polymerized adhesive. The material was packed inside the tubes, excess material was extruded and photo-polymerized for 20 s as described in Group 1.

Group 4: The discs were etched with 34.5% phosphoric acid for 15 s, rinsed for 20 s and air dried for 10 s. Silane was applied on the etched surfaces and left undisturbed for 2 min. After 2 min, the surfaces were not air-dried according to the manufacturer`s instructions

and adhesive resin (Admira bond, VOCO) was then applied for 30 s, gently air-dried for 5 s and photo-polymerized for 10 s. Two polyethylene tubes were filled with the ormocer material (shade A2), and were adjusted over the polymerized adhesive resin. The material was packed inside the tubes, excess material was extruded and photo-polymerized for 20 s as described in Group 1.

Group 5: In this group, silane was applied as in described in Group 4. Adhesive resin (Futurabond M+, VOCO) was then applied for 20 s, air-dried for 5 s and photo-polymerized for 10 s. Two polyethylene tubes were filled with the ormocer material (shade A2) and were adjusted on the polymerized adhesive resin. The material was packed inside the tubes, excess material was extruded, photo-polymerized for 20 s and stored as described in Group 1.

Group 6: Each ormocer disc was polished with silicon carbide (SiC) coated bur, supplied by the manufacturer (Cimara bur, VOCO), in one direction for 5 s. The bur was rotated at 10.000 rpm using a slow speed hand-piece (Sirona, T2 Revo-R 40, Sirona Dental System, Bensheim, Germany). The surface was air dried thoroughly and silane was applied over the polished surface and left undisturbed for 2 min. After 2 min, the surface was not air-dried according to the manufacturer`s instructions. Adhesive resin (Cimara, VOCO) was applied using the micro-brush, air spread using gentle stream of air and then left undisturbed for 20 s. The adhesive resin was photo-polymerized for 20 s. Two polyethylene tubes were filled with the ormocer material (shade A2), and were adjusted on the polymerized adhesive resin. The material was packed inside the tubes, excess material was extruded and photo-polymerized for 20 s as described in Group 1.

Group 7: In this group, the step of using the SiC bur was omitted but silane was applied for 2 min on each ground surface. After 2 min, the surface was not air-dried, and adhesive

resin (Cimara, VOCO) was applied and was photo-polymerized as in Group 6. Two polyethylene tubes were filled with the ormocer material (shade A2), and were adjusted on the polymerized adhesive resin. The material was packed inside the tubes, excess material was extruded and photo-polymerized for 20 s as described in Group 1.

2.3. Delayed repair procedure (Groups 8-14)

Ormocer discs (N=70) were prepared and stored in distilled water for 6 months. Distilled water was changed weekly until the end of storage time. After 6 months, the discs were divided into 7 groups (from Group 8 to 14, n=10 discs per group) and the repair procedures were performed similar to the immediately repaired groups.

For both immediate and delayed repair, subsequent to repair procedures, all discs with their attached ormocer micro-cylinders were stored in distilled water for 24 h prior to testing.

2.4. μ-shear bond strength test

Before μ-shear bond strength (μSBS) was tested, each polyethylene tube received two vertical cuts using surgical blade leading to the separation of each tube into two halves. Each half was removed carefully until the whole tube was removed. Excess adhesive resin or ormocer material was removed carefully from around each micro-cylinder using the surgical blade. The cylinders were examined under magnifying lens to verify the continuity of the bonding area. All examined micro-cylinders revealed no defects at the composite-composite interface and all cylinders were involved in the bond strength test.

Each ormocer disc with its bonded ormocer micro-cylinders was cemented on a rectangular shape acrylic block (10 mm x 10 mm x 70 mm) using cyanoacrylate adhesive. Each acrylic block received 4 discs, one disc on each side. The acrylic block was attached to the lower jig of the Universal Testing Machine (Lloyd instruments LR5, Fareham, UK).

An orthodontic wire of 0.2 mm diameter was wrapped around each bonded micro-cylinder as close as possible to the interface and aligned with the loading axis of the upper movable compartment of the testing machine. A shearing load with tensile mode of force was applied at a crosshead speed of 0.5 mm/min. The test was run until failure and the μSBS was calculated by dividing the load at debonding (Newton) by the bonded surface area (mm^2).

2.5. Scanning Electron Microscope (SEM)

In order to determine the effect of water storage and different surface conditioning methods on the surface topography of the material, 16 ormocer discs (2 mm x 7 mm) were prepared in the same split Teflon mold. The material was packed, photo-polymerized and divided into two main groups (8 discs each) according to the storage time: a) 24 h storage time and b) 6 months water storage. Each main storage group was further divided into four subgroups according to the surface conditioning method applied: 1) No conditioning, 2) Wet grinding using #600 grit SiC paper, 3) Wet grinding using #600 grit SiC paper and acid-etching with 34.5% phosphoric acid for 15 s, and 4) Wet grinding using #600 grit SiC paper and polishing using the corresponding bur for 5 s.

The discs were sputter coated (K550X sputter coater, Quorum Technologies Ltd, Kent, United Kingdom) and the surfaces were evaluated using the SEM (Quanta 250 FEG, FEI Company, USA) operated at 20 v and x1000 and x5000 magnifications.

2.6. Assessment of failure mode

Each tested disc was placed on a glass slide with its debonded surface exposed. The glass slide was placed under the lens of the digital microscope (Dino Capture 2.0, Dino-Lite, CA, USA) at x50 magnification. Photographs were made using the microscope. The failure modes were scored as follows: Score 1: Adhesive failure at the adhesive joint; Score 2:

Cohesive failure either in the substrate or the repair ormocer; Score 3: Mixed failure, where part of the substrate ormocer was detached or part of the repair ormocer resin composite was present on the surface of the substrate.

2.7. Statistical analysis

Statistical analyses were carried out using SPSS program version 21 (IBM Corporation, New York, USA). Two-Way ANOVA and Tukey`s tests were used where the mean μSBS values were the dependent variables, repair time (2 levels: immediate versus delayed) and surface conditioning methods (7 levels: No Conditioning, Admira Bond, Futurabond M+, Silane/Admira bond, Silane/Futurabond M+, Ceramic repair system, Silane/Cimara bond) independent variables. $P<0.05$ was considered significant in all statistical tests.

3. Results

Both the repair time (immediate vs delayed) and the surface conditioning methods showed a significant effect on the repair μSBS (MPa) of the ormocer material ($p=0.000$) (Table 2). The interaction terms between the two independent experimental factors were also significant ($p=0.000$) (2 way-ANOVA, Tukey`s).

No pre-test failures were observed. When immediate repair strengths were considered, all repair protocols tested (10.5±2.1±16.3-2.9) reached the mean bond achieved based on oxygen-inhibited layer only (no conditioning) (10.8±2.4 MPa) (Table 3). Futurabond M+ (13.9±3.4) and Silane/Cimara adhesives (16.3±2.9) showed significantly higher μSBS compared to that of the control group ($p=0.001$ and $p=0.000$, respectively).

For the delayed repair, all the bonding protocols showed significant decrease where non-conditioning (5±1.7) followed by Admira Bond (7.1±1.7) groups showed significantly lower

values compared to those of the other protocols (9.2±2.2-10.4±2.9) (p=0.000 and p=0.000, respectively).

The repair bond strength ranged from 101 to 156.7% of the original bond strength of the material for the immediate repair but the values decreased from 48.1 to 100% when delayed repair bond strength is considered.

Failure mode analysis revealed predominantly adhesive type of failures (Score 1: immediate: 95% and delayed: 90%) followed by mixed failure type (Score 3: immediate: 5% and delayed: 10%). No cohesive failures were observed either in the substrate or in the repair material (Score 2: 0%).

SEM analysis showed the presence of two different filler sizes on the specimen surfaces immediately after fabrication and after 6 months water storage, the fillers became more evident (Figs. 2a-b). The specimens immediately after grinding showed the presence of obvious grinding grooves with the formation of smear layer but after 6 months water storage, less grinding grooves but more exposure of fillers was evident (Figs. 3a-d). Specimen surfaces immediately after grinding and etching showed less grinding grooves and most of the grinding smear layer was removed after acid etching compared to the grinded specimens only. In the same group, after 6 months water storage, no change was observed in the ormocer surface compared to the water stored grinded specimens (Figs. 4a-d). In the silicone carbide bur grinded specimens, no grinding grooves were evident, and after 6 months water storage, relatively smooth surface with some surface debris was observed (Figs. 5a-d).

4. **Discussion**

The need for minimal intervention of defective restorations is gaining more attention in dentistry. Repair of an existing restoration offers many advantages, [11] and not only shortens the restorative treatment time but also offers a conservative treatment approach, [7,11] and extends the life span of the restoration. [12] This study evaluated the repair bond strength of newly introduced ormocer material as a function of different repair protocols and repair time. Since both parameters studied showed a significant effect on the repair bond strength the null hypothesis could be rejected.

Although bisphenol-A Glycidyl Methacrylate (bis-GMA) monomer is widely used in commercial resin composites, the release of bisphenol-A from the resinous matrix could increase its cytotoxic behaviors. For this reason, ormocer material was lunched in the market with a matrix of less polymerization shrinkage and less or no cytotoxicity compared to the traditional dimethacrylate resins. This kind of material does not contain free dimethacrylate monomers but its resin terminates with C=C group. [13] Several previous studies revealed comparable [3,13,14] or even better [15] results with ormocer based material compared to the traditional methacrylate composites.

One of the important properties of successful restorative dental materials is their repair potential either immediately or after their service in the oral cavity. [1,3] Bond strength of repaired restoration depends on numerous factors such as substrate surface condition, storage time, storage media before and after repair, presence and composition of intermediate agent, chemical microstructure of the substrate and repair material. [1,3-5] The key factor for the success of resin composite repair is the high bond strength between the previously polymerized composite and the freshly added one. [16] Due to improper handling of the resin composite material, incorrect matrix application or the inappropriate

finishing and polishing procedures, [1] the formation of surface voids, under contours or sub-margins at restoration/tooth interface raise the need for immediate repair of resin composite restorations. [17] On the other hand, the presence of discolored margin, marginal ditching or minimal fracture might also necessitate the repair of aged restorations. [18] Surface grinding was suggested for all repair procedures in order to create a rough surface and improve the repair bond strength of resin composite in previous studies. [2,19-22] Mechanical surface treatment is a mandatory step to remove defected tooth tissues that also cleans surface of resin composite and helps to bond the new layer of material to the aged one.

Since the new ormocer material does not contain free dimethacrylate monomers, [13] one could expect that the bond between the newly added material and the pre-polymerized one could be compromised. The use of matrix on the unpolymerized resin material prevents the formation of the viscous and the partially polymerized oxygen inhibited layer. [1,23,24] As claimed by the manufacturer, this material, in spite of the fact that it does not contain traditional methacrylate resin, when polymerized, the superficial surface layer will be an oxygen inhibited layer that contains unreacted C=C groups that will allow for bonding between the different layers (Research and Development Department, VOCO). Accordingly, an additional group was included to repair the ormocer material, when its surface was polymerized in atmospheric air that received no surface conditioning. [25] From the results of this study after direct repair, when ormocer resin composite was photo-polymerized in air, the repair bond strength was not statistically significant from the groups that were polymerized in the presence of matrix (i.e. in the absence of the oxygen inhibition layer). The significance of the oxygen inhibition layer on either the immediate repair or bonding between resin composite layers is still controversial. [26] The immediate repair bond strength was improved [27] and bonding between resin composite increments was

enhanced in the absence of the oxygen inhibition layer. [28] In previous studies, immediate repair bond strength of different resin composites was not affected by the presence or absence of the oxygen inhibition layer, [14,26] which was in agreement with the results of this study. On the contrary, the presence of oxygen inhibition layer was crucial to improve the immediate repair bond strength of the micro-fine hybrid resin composite [17] which is in disagreement with the results of this study. Thus, it could be concluded that the presence of oxygen inhibition layer to improve bonding between ormocer layers was not crucial, and the bonding between increments could be maintained even in the absence of the oxygen inhibition layer.

Two intermediate bonding agents of the corresponding resin composite, as recommended by the manufacturer, were used in this study. Admira bond was a simplified etch-and-rinse ormocer-based adhesive resin, while Futurabond M+ was a self-etching, single-bottle adhesive. Both adhesives, as recommended by the manufacturer, could be used to bond the ormocer material to both enamel and dentin. Futurabond M+ improved the immediate repair bond strength, and this improvement could be attributed to the presence of obvious grinding grooves that acted as mechanical retentive sites into which the self-etching adhesive interlocked after polymerization. [17] This result was in contrast with a previous study [2] where the use of the intermediate adhesive resin did not improve the immediate repair bond strength. This could be attributed to the fact that in that study, the intermediate adhesive resins were applied on smooth unground surfaces, while in the current study the adhesive was applied over grinded surfaces. When an adhesive resin was applied on smooth surfaces, the immediate repair bond strength was not statistically improved. On the other hand, when the same adhesive resin was applied on grinded surfaces, the immediate bond strength was statistically enhanced. [17] It was reported that the presence of such rough and irregular resin composite surface might be advantageous for the mechanical

retention into which the applied adhesive can diffuse. [17]

Although Admira bond is ormocer based adhesive resin, its application did not improve the repair bond strength and showed no statistical significant difference compared to the unconditioned groups. The application of Admira bond was preceded with the application of acid etching as recommended by the manufacturer. Repair of resin composites could include the conditioning of both tooth substrates, removal of defected tissues and part of the resin composite restoration. [18] In this situation, when etch-and-rinse adhesives were used, acid etching could contaminate the surface of resin composite, especially if the defect is small. As previously reported, the application of acid etching did not change the morphology of the grinded surfaces and its action was limited to cleaning the surfaces from smear debris and grinding dust. [29,30] On the other hand, one study [17] showed that the action of acid etching was not only limited to its cleaning effect as it changed the morphology of the grinded resin composite surface to less obviously rough pattern. SEM finding of this study showed that acid etching removed the smear layer that was created during surface grinding and the surface appeared with relatively lesser grinding pattern. It could be hypothesized that the application of acid etching enhanced the wettability of the resin composite surface that eventually enabled the chemical adhesion between the newly added material, through the intermediate adhesive layer, and the previously polymerized one. [17] This hypothesis was used to explain the improvement in immediate repair bond strength, only when acid etching was applied on ungrinded resin composite surface. Although, Admira bond is an ormocer based resin adhesive, mechanical interlock might play the major role in the improvement of the immediate repair. Admira bond was applied passively for 30 s and gently air dried to remove excess solvent. This adhesive is acetone based adhesive and acetone has a high vapor pressure that evaporates rapidly from the surface [31] Rapid evaporation of solvent combined with the extended time of application

might lead to the formation of viscous adhesive layer. Increase in adhesive viscosity might lead to the inability of the adhesive to diffuse properly through the mechanical retentive sites created by the grinding procedure.

The effect of silane on the immediate repair bond strength of a nano-hybrid resin composite was discussed in a recent study. [18] The authors concluded that silane showed no effect on the immediate repair bond strength, regardless of the resin composite surface condition. In the current study, silane did not improve the immediate repair bond strength of the ormocer material that in agreement with this study. In this study, silane was added and left to react with the surface for 2 min and it was not air dried as recommended by the manufacturer. This might allow the silane to diffuse through the ground surface and prevent further diffusion of the adhesives. When both silane and adhesive resin were applied separately in two consecutive steps, this allowed to the formation of thick and multiphase layers at the interface that could weaken the bond. [30] In addition, silane might react with the hydroxyl groups of the glass fillers, in the presence of water, and the reaction could lead to the formation of highly cross-linked siloxane group. [33] This reaction could play a role in the chemical bond between the freshly added resin composite, through the adhesive layer, and the already polymerized one. The reaction might not be achieved if the surface of resin composite was dry. [18] As, the surface of the ormocer material in this study was air dried before the application of silane, the reaction of silane with the surface of glass fillers might not be expected. Accordingly, the use of silane could not be beneficial, before the application of the intermediate adhesives, to immediately repair the material.

Recently, different repair systems are advised to improve the repair bond strength of aged resin composite. [34] Cimara repair kit is a universal repair system that can be used to repair both ceramics and indirect resin composite materials. The silicon carbide bur (SiC

bur) supplied by the manufacturer was used for surface polish after grinding with the abrasive tool. As suggested by the manufacturer, this step could be omitted when the repair is performed on resin composite materials. As to our knowledge, the repair potential of Cimara repair kit was not evaluated with and without the use of the SiC bur. Cimara bond was applied on the ormocer surface and gently spread using weak stream of compressed air and then it was left undisturbed to act on the surface for 20 s. As the air-drying step was omitted for Cimara adhesive, this might lead to a relatively thick adhesive layer on the surface compared to the adhesive layer formed by Admira bond and Futurabond M+. This relatively thick layer might act as stress absorber during shear test, improving the repair bond strength. The use of SiC bur of the ceramic repair system showed no improvement in the repair bond strength, compared to the unconditioned groups. It should however be noted that resin composite surface was ground using #600 SiC paper. This resulted in a rough surface with obvious grinding grooves with the deposition of SiC particles and remnants detached from SiC particles on the substrate surface. The deposition of SiC particles blocks the grinding grooves that acted as retentive sites for the adhesive. The formation of the relatively thick smear layer of SiC particles could result in bonding of the newly added resin composite through the aid of the intermediate adhesive layer to the created smear layer. As the smear layer is loosely attached to the surface, the bond strength could not be expected to exceed the bond strength between the smear layer and the surface onto which it was formed. [35]

For the delayed repair bond strength, all repair protocols improved the bond strength between old and new ormocer materials compared to the unconditioned control group, with the lowest results for Admira Bond. In previous study, the use of intermediate adhesive layer improved the repair bond strength of the microfilled resin composite. [36] Furthermore, surface conditioning in the form of grinding with [37] and without [38,39]

adhesive application significantly improved the repair bond strength of a microfilled resin composite. On the contrary, in another study, [40] the repair of aged micro-fine hybrid composite was not improved with the application of intermediate adhesive, even with surface grinding.

Application of silane showed contradictory results in this study. Silane application prior to the application of Admira bond improved the bond strength, in contrast to Futurabond M+, which showed no improvement in bond strength with silane application. SEM micrographs indicated that surface grinding of aged specimens did not produce the same morphological feature as did in immediate specimens. Grinding of aged specimens did not produce grooves and its action was limited to exposure of inorganic fillers where some fillers were partly detached from the surface. This contradictory results might be attributed to the fact that silane was applied after acid etching for 15s. Acid etching cleaned the surface and might have rendered the reaction of exposed fillers with the silane. It could be anticipated that the surface of aged ormocer composite was cleaned with acid etching prior to the application of silane that needs further investigations as Admira bond was the only bond applied as an etch-and-rinse adhesive in this study. These results, except Admira result, was in agreement with a previous study where silane did not add any significant improvement in the repair bond strength of the aged resin composite. [18] On the contrary, the results with Admira bond were in agreement with another study [40] regardless of the difference in the resin composites used.

Regarding the comparison between the immediate and delayed repair bond strength, immediate repair bond strength showed significantly higher bond strength within all bonding protocols. SEM micrographs showed that when the ormocer material was stored for 6 months in water, exposure of the fillers on the material surface became more evident.

This could be a result of surface degradation of the ormocer material that impairs the repair bond strength. [40]

Grinding water stored discs did not change the surface morphology compared to the immediate discs. Grinding of immediately prepared discs produced grinding grooves with evident smear layer. Nonetheless, grinding of water stored discs exposed limited fillers on the surface and detached some fillers from the surface. As mentioned above, the presence of grinding grooves could have acted as retentive sites for the attachment of adhesive resin after polymerization and thus improved bond strength. [17] The lack of these grinding grooves might be the reason for the drop in the repair bond strength of the stored specimens, regardless to the bonding protocol used. The lack of grinding grooves, deprived the surface from its mechanical attachment needed for the improvement of the bond strength, which made the bonding between the prepolymerized material and the newly added one limited to chemical bond only. As this type of material does not have residual attachment sites due to complete consumption of the monomer after polymerization of the material, as claimed by the manufacturer, they rely on the chemical bonding only, which was not enough to improve the bond strength.

5. Conclusions

From this study, the following conclusions could be drawn:

(1) For immediate repair, 24 h after fabrication of ormocer with the same material, either silane coupling agent followed by adhesive resin application (Cimara) or adhesive resin only (Futurabond M+) could be suggested.

(2) The delayed repair bond strength of ormocer after 6 months could be improved with any

bonding protocol used in this study, except for Admira bond.

(3) The evidence of chemical bond between the pre-polymerized ormocer material and the freshly added layer was not verified in this study.

(4) Failure modes were mainly adhesive after both immediate and delayed repair and no cohesive failures were observed either in the substrate or in the repair material in any of the repaired specimens.

Clinical relevance

The use of self-etching adhesive resin or ceramic repair kit could be advised for immediate repair of the new ormocer material. For delayed repair, except for Admirabond, any bonding protocol employed in this study could be used, providing that adhesive failure types were more predominant and no cohesive failures were observed after any of the repair protocol.

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Conflict of interest

The authors did not have any financial or commercial interest in any of the materials used in this study.

REFERENCES

[1] Rinastiti M, Özcan M, Siswomihardjo W, et al. Immediate repair bond strengths of microhybrid, nanohybrid and nanofilled composites after different surface treatments. J. Dent. 2010;38:29-38.

[2] Cramer NB, Stansbury JW, Bowman CN. Recent advances and developments in composite dental restorative materials. J. Dent. Res. 2011;90:402-416.

[3] Kalra S, Singh A, Gupta M, et al. Ormocer: An aesthetic direct restorative material; An in vitro study comparing the marginal sealing ability of organically modified ceramics and a hybrid composite using an ormocer-based bonding agent and a conventional fifth-generation bonding agent. Cont. Clin. Dent. 2012;3:48-53.

[4] Tezvergil A, Lassila L, Vallittu P. Composite–composite repair bond strength: effect of different adhesion primers. J. Dent. 2003;31:521-525.

[5] Cavalcante LM, Schneider LF, Silikas N, et al. Surface integrity of solvent-challenged ormocer-matrix composite. Dent. Mater. 2011;27:173-179.

[6] Awad D, Ilie N. Effect of polymerisation and ageing on the incremental bond strength of ormocer-based dental materials. Clin. Oral Invest. 2013;17:1339-1347.

[7] Hickel R, Brüshaver K, Ilie N. Repair of restorations--Criteria for decision making and clinical recommendations. Dent. Mater. 2012;29:28-50.

[8] Jafarzadeh Kashi TS, Erfan M, Rakhshan V, et al. An in vitro assessment of the effects of three surface treatments on repair bond strength of aged composites. Oper. Dent. 2011,36:608-617.

[9] Baur V, Ilie N. Repair of dental resin-based composites. Clin. Oral Invest. 2013;17:601-608.

[10] Magni E, Ferrari M, Papacchini F, Hickel R, Ilie N. Influence of ozone application on the repair strength of silorane-based and ormocer-based composites. Am. J. Dent. 2010;23:260-264.

[11] Lynch CD, Hayashi H, Seow LL, et al. The management of defective resin composite restorations: Current trends in dental school teaching in Japan. J. Oral Rehabil. 2013;38:497-504.

[12] Gordan VV, Garvan CW, Blaser PK, et al. A long-term evaluation of alternative treatments to replacement of resin-based composite restorations: results of a seven-year study. J. Am. Dent. Assoc. 2009;140:1476-1484.

[13] Al-Ahdal K, Silikas N, Watts DC. Development of viscoelastic stability of resin-composites incorporating novel matrices. Dent. Mater. 2015;31:1561-1566.

[14] Mahmoud SH, El-Embaby AE, Abdallah AM, et al. Two-year clinical evaluation of ormocer, nanohybrid and nanofill composite restorative systems in posterior teeth. J. Adhes. Dent. 2008;4:315-322.

[15] Civelek A, Ersoy M, L'Hotelier E, et al. Polymerization shrinkage and microleakage in Class II cavities of various resin composites. Oper. Dent. 2003;28:635-41.

[16] Özcan M, Pekkan G. Effect of different adhesion strategies on bond strength of resin composite to composite-dentin complex. Oper. Dent. 2013;38:63-72.

[17] El-Askary FS, Fawzy AS, Abd Elmohsen HM. Tensile bond strength of immediately repaired anterior microfine hybrid restorative composite using nontrimmed hourglass specimens. J. Adhes. Dent. 2009;11:41-47.

[18] El-Askary FS, El-Banna AH, van Noort R. Immediate vs delayed repair bond strength of a nanohybrid resin composite. J. Adhes. Dent. 2012;14:265-274.

[19] da Costa TR, Serrano AM, Atman AP, et al. Durability of composite repair using different surface treatments. J. Dent. 2012; 40:513-521.

[20] Costa TR, Ferreira SQ, Klein-Júnior CA, et al. Durability of surface treatments and intermediate agents used for repair of a polished composite. Oper. Dent. 2010;35:231-237.

[21] Turner CW, Meiers JC. Repair of an aged, contaminated indirect composite resin with a direct, visible-light-cured composite resin. Oper. Dent. 1993;18:187-194.

[22] Chiba K, Hosoda H, Fusayama T. The addition of an adhesive composite resin to the same material: Bond Strength and clinical techniques. J. Prosth. Dent. 1989;61:669-675.

[23] Truffier-Boutry D, Place E, Devaux J, et al. Interfacial layer characterization in dental composite. J. Oral Rehab. 2003;30:74-77.

[24] Rinastiti M, Özcan M, Siswomihardjo W, et al. Effects of surface conditioning on repair bond strengths of non-aged and aged microhybrid, nanohybrid, and nanofilled composite resins. Clin. Oral Invest. 2011;15:625-633.

[25] Özcan M, Barbosa S, Melo R, et al. Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. Dent. Mater. 2007;23:1276-1282.

[26] Dall'Oca S, Papacchini F, Goracci C, et al. Effect of oxygen inhibition on composite repair strength over time. J. Biomed. Mater. Res. Part B: App. Biomater. 2007;81:493-498.

[27] Lewis G, Johanson W, Martin W, et al. Shear bond strength of immediately repaired ligh-cured composite resin restorations. Oper. Dent. 1998;23:121-127.

[28] Eliades G, Caputo A. The strength of layering technique in visible light-cured composites. J. prosth. Dent. 1989,61:31-38.

[29] Hannig C, Laubach S, Hahn P, et al. Shear bond strength of repaired adhesive filling materials using different repair procedures. J. Adhes. Dent. 2006;8:35-40.

[30] Papacchini P, Dall'Oca S, Chieffi N, et al. Composite-to-composite microtensile bond strength in the repair of microfilled hybrid composite: effect of surface treatment and oxygen inhibition. J. Adhes. Dent. 2007;9:25-31.

[31] Van Landuyt KL, Snauwaert J, De Munck J, et al. Systematic review of the chemical composition of contemporary dental adhesives. Biomaterials. 2007; 28:3757-3785.

[32] Park S, Jin J. Effect of silane coupling agent on interfase and performance of glass fibers/unsaturated polyester composite. J. Col. Interface. Sc. 2001;242:174-179.

[33] Özcan M, Matinlinna J, Vallittu P, et al. Effect of drying time of 3-mathacryloxypropyltrimethoxy silane on the shear bond strength of a composite resin to silica-coated base/noble alloys. Dent. Mater. 2004;20:586-590.

[34] Ilie N, Stawarczyk B. Efficiency of different repair kits on bonding to aged dental resin composite substrates. Inter. J. of Adhes. and Adhes. 2015;58:7-12.

[35] El-Askary FS, Nassif MS, Fawzy AS. Shear bond strength of glass-ionomer adhesive to dentin: Effect of smear layer thickness and different dentin conditioners, J. Adhes. Dent. 2008;10:471-479.

[36] Azarbal P, Boyer D, Chan K. The effect of bonding agents on the interfacial bond strength of repaire composites. Dent. Mater. 1986;2:135-155.

[37] Jafarzadeh Kashi TS, Erfan M, Rakhshan V, et al. The effects of three surface treatments on repair bond strength of aged composites. Oper. Dent. 2011;36:608-617.

[38] Shahdad SA, Kennedy JG. Bond strength of repaired anterior composite resins: an in vitro study. J. Dent. 1998;26:685-694.

[39] Rathke A, Tymina Y, Haller B. Effect of different surface treatments on the composite–composite repair bond strength. Clin. Oral Invest. 2009;13:317-323.

[40] Fawzy AS, El-Askary FS, Amer MA. Effect of surface treatments on the tensile bond strength of repaired water-aged anterior restorative micro-fine hybrid resin composite. J. Dent. 2008;36:969-976.

Captions to the legends and tables:

Tables:

Table 1. Materials, types, chemical compositions (Batch #) and manufacturers.

Table 2. Results of two-Way ANOVA and Tukey`s tests for the effect of repair time, surface conditioning methods and their interactions on the repair bond strength of ormocer material.

Table 3. Means ± Standard Deviations for the effect of repair time within each surface conditioning method and the effect of surface conditioning within each repair time on the repair bond strength of the ormocer material. Means with same superscript small letters within each column are not statistically significant at P>0.05.

Figures:

Fig. 1 Flow chart showing the allocation of experimental groups.

Figs. 2a-b SEM micrographs of non-conditioned specimens **a)** specimen surface immediately after fabrication showing the presence of two different filler sizes (x5000), **b)** specimen surface after 6 months water storage showing that the fillers became evident on the ormocer surface (x5000).

Figs. 3a-d SEM micrographs of grinded specimens **a)** specimen surface immediately after grinding showing the presence of obvious grinding grooves with the formation of smear layer over the ormocer surface (x1000), **b)** higher magnification of a, (x5000), **c)** specimen surface after 6 months water storage revealing the absence of the grinding grooves with the exposure of fillers (x1000), **d)** higher magnification of c (x5000).

Figs. 4a-d SEM micrographs of the grinding and acid-etched specimens **a)** specimen surface immediately after grinding and etching showing that the grinding grooves appeared with less distinct pattern (x1000), **b)** higher magnification of a revealing relatively clean ormocer surface and most of the grinding smear layer was removed due to the acid etching compared to the grinded specimens only (x5000), **c)** specimen surface after 6 months water storage showing no change in the ormocer surface compared to the water stored grinded specimens (x1000), **d)** higher magnification of c (x5000).

Figs. 5a-d SEM micrographs of the silicone carbide bur grinded specimens **a)** specimen surface immediately after bur grinding, showing no evident grinding grooves (x1000), **b)** higher magnification of a, revealing the presence of relatively smooth surface due to the formation of smear layer over the surface after polishing with the bur (x5000), **c)** specimen surface after 6 months water storage showing a relatively smooth surface (x1000), **d)** higher magnification of c, revealing the presence of surface debris that might be due to the polishing procedure with the bur (x5000).

Table 1. Materials, types, chemical compositions (Batch #) and manufacturers.

Materials	Chemical Composition (Batch#)	Manufacturer
Admira Fusion (Ormocer Resin Material)	Ormocer Resin, CQ, amine, BHT, SiO_2 nano particles (20 - 40 nm), glass ceramics (1 µm), iron oxide, titanium dioxide. Filler content: 84 %w/w = 69% vol% (V56861)	VOCO GmbH, Cuxhaven, Germany
Admira bond (Etch-and-Rinse Adhesive)	Vococid acid etching gel: 34.5% phosphoric acid etching gel (1411479) Bond: Ormocer resin, dimethacrylates, HEMA, NaF, acid modified methacrylates, CQ, BHT, acetone (1421529)	VOCO GmbH
Futurabond M+ (Self-etch Adhesive)	Dimethacrylates, fumed silica, acid modified methacrylates, camphorquinone, BHT, amine, ethanol, water (1414311)	VOCO GmbH
Cimara (Adhesive Resin)	Acetone, dimethacrylates, carbon acid modified dimethacrylates, CQ, BHT, Amine (1414216)	VOCO GmbH
Silane Coupling Agent	Reactive silane, isopropanol, acetone, amine (1415052)	VOCO GmbH

Table 2. Results of two-Way ANOVA and Tukey`s tests for the effect of repair time, surface conditioning methods and their interactions on the repair bond strength of ormocer material.

Source	Type III Sum of Squares	df	Mean Square	F	Significance
Corrected Model	1940.490[a]	13	149.268	25.951	0.000
Intercept	31122.514	1	31122.514	5410.856	0.000
Repair time (Immediate vs Delayed)	986.626	1	986.626	171.532	0.000
Surface conditioning method	762.549	6	127.091	22.096	0.000
Repair time * Surface conditioning	191.315	6	31.886	5.544	0.000
Error	1529.996	266	5.752		
Total	34593.000	280			
Corrected Total	3470.486	279			

Table 3. Means ± Standard Deviations for the effect of repair time within each surface conditioning method and the effect of surface conditioning within each repair time on the repair bond strength of the ormocer material. Means with same superscript small letters within each column are not statistically significant at P>0.05.

	Immediate repair	**Delayed repair (6 months)**	**P value**
No conditioning	10.8±2.4^{c}	5±1.7^{c}	0.000
Admira Bond	10.5±2.1^{c}	7.1±1.7^{b}	0.000
Futurabond M+	13.9±3.4^{ab}	9.2±2.2^{ab}	0.000
Silane/Admira bond	12±2.6^{bc}	9.4±1.7^{a}	0.001
Silane/Futurabond M+	11±1.8^{c}	9.3±2.2^{a}	0.011
Ceramic repair system	12.5±2.5^{bc}	10.4±2.9^{a}	0.022
Silane/Cimara bond	16.3±2.9^{a}	10.2±2.6^{a}	0.000